International Journal of Mechanical and Production Engineering Research and Development (IJMPERD)
ISSN (P): 2249–6890; ISSN (E): 2249–8001
Vol. 11, Issue 4, Aug 2021, 367-378

EVOLVING NEW SPECIFICATION AND DEVELOPMENT OF FLEXIBLE RUBBER DIAPHRAGM OF AN AIRCRAFT WITH GRAPHENE FILLED NITRILE RUBBER NANO-COMPOSITE

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ABSTRACT

The flexible rubber diaphragm (FRD) fails prematurely during service exploitation on aircraft due to various reasons. Efforts were made to review the qualification requirements of existing rubber compound specifications. Accordingly, new technical specification evolved for indigenous development, evaluation and airworthiness certification. End use criticality of FRD and longevity in terms of flying hours of aircraft was a major input considered while evolving the new technical specification along with long lasting in terms of calendar years of life. As per evolved technical specification, a new rubber-nano-composite was designed & developed. Graphenenanoplatelet is one of the performance enhancement ingredients used in the nitrilebased rubber nano-composites in addition to conventional compounding ingredients. Optimisation of graphenenanoplatelet loading was considered necessary mainly to improve fatigue properties, air permeability resistance and thermal ageing along with low rejection during manufacturing of FRD.

KEYWORDS: Evolving , Flexible Rubber , Aircraft , Graphene Filled Nitrile Rubber Nano-Composite

Received: Jul 01, 2021; **Accepted**: Jul 21, 2021; **Published**: Jul 31, 2021; **Paper Id**.: IJMPERDAUG202128

Original Article

1. INTRODUCTION

To ensure sovereignty of a country, Air power is an important aspect and it is mainly being achieved through the use of a variety of aircraft [1]. Defence aircraft play an important role in air defence and requires a high degree of performance throughout various missions, which begins from take-off and ends with safe landing. Therefore, the performance of each and every system, sub-system, equipment, rubber components and other structural parts of an aircraft is vital. It is a well known fact that aircraft and its components, materials particularly rubber compounds etc. fall under the proprietary item category and minimal design details, technical specifications etc. are provided by the country of origin {i.e. Original Equipment Manufacturer (OEM) of Aircraft/components}. This minimal technical information for manufacturing and quality control test parameters and batch acceptance criteria is not enough to judge the quality as well as performance related confirmation of the final product. The material of flexible rubber diaphragm (FRD) also falls under this category and the bare minimal requirement of existing OEM14 rubber compound provided for manufacturing of FRD is specified and outlined in the existing technical specification [2, 3].This diaphragm is generally manufactured using nitrile based rubber compound to use in hydraulic fluid of aircraft. But, it was noticed that these nitrile rubber based diaphragms were not performing as expected [4]. There are various reasons behind the failure of a diaphragm during the service uses of the aircraft [4]. The present work is

intended to develop a new technical specification for FRDs with improvement to meet the expected performance.

2. EXISTING MATERIAL OF FLEXIBLE RUBBER DIAPHRAGM

The Existing rubber compound used for manufacturing of the flexible rubber diaphragm (FRD) is identified as OEM14 and presently imported from OEM and under license manufacturing.

1.1 Existing Technical Specification and Requirement Analysis:

The requirements specified in the existing technical specification [2, 3] of OEM14 rubber compound used for making of flexible rubber diaphragm (FRD) have been studied in details. On studying the contents of the technical specification [2, 3]; it was observed that the requirements specified appear to be mainly for quality control and batch acceptance of rubber compound. The physical-mechanical properties stipulated in the existing specification [3] for the existing rubber compound (OEM14) are given in table 1. In addition to properties mentioned in table-1, storage requirement related information is specified in the specification [3, 5]. The rubber compound should be stored in a dimly light room with temperature from -5 to 25^{0}C and with a relative humidity of not more than 85% [3, 5].

Table 1: Technical requirement of existing rubber compoundOEM14 [3]

S No.	Test parameter	Requirement
1.	Tensile strength (MPa) (min)	11
2.	Relative elongation at break (%) (min)	160
3.	Hardness (Shore A)	72-79
4.	Ageing co-efficient at 70^{0}C for 144 hrs (min)	0.6
5.	Volumetric swelling(%)in hydraulic fluid FH-51 at 70^{0}C for 24 hrs	2 to 12
6.	Gravimetric swelling (%) inhydraulic fluid FH-51at 70^{0}C for 24 hrs	- 1 to + 8
7.	Relative residual deformation (%)@ 30% defln./100^{0}C/70 hrs (max)	60
8.	Brittleness test temperature^{0}C	-38
9.	Coefficient of frost resistance at -45^{0}C / 30 %compression (min)	0.15
10.	Operating temperature: Air Hydraulic fluid FH-51	-45 to +100^{0}C -60 to +100^{0}C
11.	Shrinkage (%)	1.6 -1.8
12.	Density (gm/cm^3) * record value	1.28*
13.	Curing temperature&duration	143+1^{0}C&40 min
Base: Nitrile **Use**: Manufacturing of Flexible rubber diaphragm (FRD), Rubber bonded valves and seals etc.		

2. Life of Flexible Rubber Diaphragm

The FRD manufactured using OEM14 was not lasting for full assigned life (i.e. 1000flying hrs/10 year's life [6, 7].) and had failedprematurely due to various reasons. The long duration of usage of FRD should be without any performance degradation in the prevailing operating conditions of aircraft.

Meanwhile, attempts have been made for the indigenous development of rubber compounds to manufacture FRD and overcome service failures. Accordingly, technical specification was evolved [5]. Indigenous rubber compound NPE 8 [6] was developed and evaluated as per the requirement specified in the specification [1, 2, 5]. However, the FRD made from indigenous rubber compound NPE8 is also not able to withstand the number of cycles specified in the endurance test document [8].The FRD made from rubber compound NPE8 is able to withstand only half the number of endurance cycles during testing and assigned half-life (i.e. 500 hrs in place of required life of 1000 hrs) only [6].

Therefore, it was imperative to design and indigenously develop the "rubber-nano-composite" using grapheme nano-platelets for manufacturing the flexible rubber diaphragm (FRD). The FRD manufacture using newly developed rubber-nano-composite has been designed to withstand the full endurance cycles [8] and is also expected to have the same life as specified for existing FRD made from OEM14 (i.e. 1000flying hrs/10 years) [6, 7].

3 REVIEW OF SPECIFICATION

3.1 Physical-Mechanical Properties

Physical-Mechanical Properties outlined in table 1have been analysed, discussed and revised as mentioned in succeeding paragraphs below:

3.1.1 Tensile Properties

Tensile strength and elongation at break are in general, useful for rubber compound design development, optimisation of compounding ingredients and assessing the rubber compound's vulnerability due to various degradations over the period of time. Tensile properties are also excellent quality controlling parameters to monitor the consistency of product's quality, once the rubber compound is successfully developed. These tests are sensitive to changes in manufacturing conditions and even used to identify under or over vulcanization, improper mixing of ingredients and the presence of foreign matter etc.

Tensile properties are also useful to adjudge the usefulness of a particular rubber.The measure of tensile properties before and after exposure to heat, fluids, ozone, weathering etc. assist in determining the relative resistance of group of rubber compounds. Even a small amount of deterioration indicates appreciable changes in tensile properties especially elongation at break. The superior retention of elongation at break after ageing is a good indication that the rubber will retain most of its other properties over a period of time.

However, tensile properties are mainly for grading of particular rubber compound for a particular application and not having direct implications on the performance of any Rubber product such as flexible rubber diaphragm (FRD). Therefore, the tensile properties specified for existing rubber compound OEM14 [3] suffice for the intended critical end use of FRD on spherical hydraulic accumulator of typical aircraft.So, no change was proposed on tensile properties.

3.1.2 Hardness

Hardness is the resistance to penetration or deformation of material surface on application of indenting applied load and represents the elastic modulus of the material. In other words, it indicates the stiffness which is reciprocal of flexibility of the component. Rubber product has inherent flexibility and greatly influences by hardness [9, 10]. Hardness of a rubber component mainly depends on the hardness of base polymer, amount of fillerloading along with other compounding ingredients and also extent of vulcanisation. Considering the end use of the flexible rubber diaphragm, hardness should be reasonably low as compared to the other rubber products like seals, rubber bonded valves etc. Lower degree of the hardness mainly facilitates the flexibility and enhances life (i.e. in terms of number of cycles) of the flexible rubber diaphragm.

Considering the end use application of FRD, the hardness (72-79 Shore A) as per existing specification [3] was found to be very high and that could be one of the reasons for premature failure. In fact during the service; these diaphragms continuously remain under flexing and amount depends mainly on extent of pressure fluctuation in the hydraulic system of the aircraft. The diaphragm experiences the highest deflection once the engine driven pump (EDP) of

aircraft develops maximum hydraulic pressure in the system. Once the hydraulic pressure reduces (due to operation of various systems, sub-systems and use in other services of aircraft); fully deflected flexible rubber diaphragm will attend intermediate position.

Figure 1 (a) & (b) depict the minimum and maximum deflected position of flexible rubber diaphragm respectively. Under the maximum defection condition; the diaphragm experiences highest stress concentration through the circumferential edge, which is very sever. The repetitive flexing and sever deflection of diaphragm at higher rate, definitely damages the rubber material especially at the circumferential edge regions and lead to initiation of the crack formation etc. This type of phenomena (i.e. crack initiations at the circumferential edge regions) is further aggravated with higher hardness as compared to the lower hardness of the flexible diaphragm due to inherent material characteristics.

In fact, Fatigue is an appropriate term for repetitive cyclic deformation of flexible diaphragm taking place during service usage on the aircraft. Repetitive cyclic deformation of FRD mainly and significantly affects the stiffness along with loss in mechanical strength and ultimately leads to breakdown. The fatigue of flexible rubber diaphragm may also enhance thermal degradation, oxidation leads to cracks formation and finally tearing of component. The type of degradation during fatigue varies according to the geometry of FRD and intensity of stressing conditions.

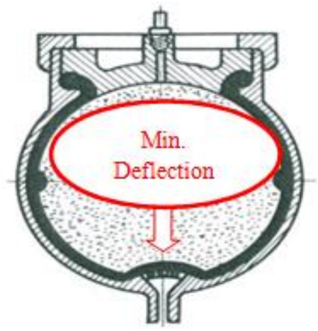

Fig. 1(a): Minimum deflection position of FRD

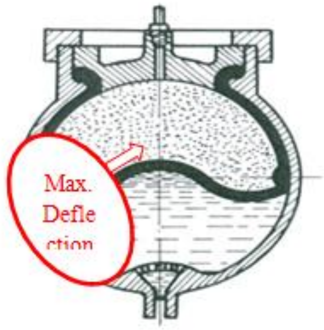

Fig. 1(b): Maximum deflection position of FRD

Considering the above stated findings and in-depth characterisation along with establishing the causes of failure of flexible diaphragm; it is imperative to review and lower the hardness requirement of FRD rubber compound. Therefore, the harness value specified for OEM14 in the range 72 to 79 Shore A has been reviewed and lowered to 60±5 Shore A [9, 10].

3.1.3 Thermal Air-Ageing

The flexible rubber diaphragm (FRD) has to last for long 10 years in terms of calendar life without significant degradation in performance is an important requirement plus 5 years in storage [6,7]. During service exploitation, significant degradation in various rubbery properties takes place in few days to several years depending on end use and environmental conditions of component. Moreover, appreciable degradation during storage which is 5 years [6, 7] is added even though the component is stored as per specified conditions. In order to expedite degradation, thermal ageing is one of the useful techniques and it evolved over a period of time. Therefore, thermal air ageing requirement specified for OEM14 in the existing technical specification has been reviewed and studied. It is observed that the test conditions of air ageing specified

in the existing technical specification is at 70^0C for 144hrs and permitted degradation ascertained by ageing coefficient is 0.6 [ref table 1] appears to be inadequate. Whereas the flexible rubber diaphragm (FRD) has to last for long 10 years at continuous temperature of about 100^0C during service exploitation is quite severe [6, 7]. Therefore, air ageing degradation conditions and acceptable level of ageing coefficient is imperative to revise. In this regard, inputs from a long term natural ageing report by RC Moakes (11) is very vital. Considering the end use criticality of RFD and inputs from ISO [12] also taken into consideration while revising air ageing requirement. Accordingly, air ageing temperature revised to 90^0C [11] (in place of 70^0C) for a duration of 336 hrs (in place of 144 hrs) with minimum ageing coefficient of 0.5 in place of 0.6[13] is appropriate to ascertain the longevity to meet the long end use of FRD. The revised air ageing requirements have been included in the new evolved technical specification.

5.1.4 Compression Set, Relative Residual Deformation

Compression set, relative residual deformation property of existing rubber compound OEM14 specified in the existing technical specification [3] has been studied and analysed. The compression set specified for OEM14 with 30 % compression at 100^0C for 72hrs is 60% (max); which appears to be on the higher side and relaxed qualification requirement especially for collar portion of flexible rubber diaphragm as shown in fig.1 (a) & (b). Collar portion of flexible rubber diaphragm also acts as a sealing ring to prevent nitrogen leakage.

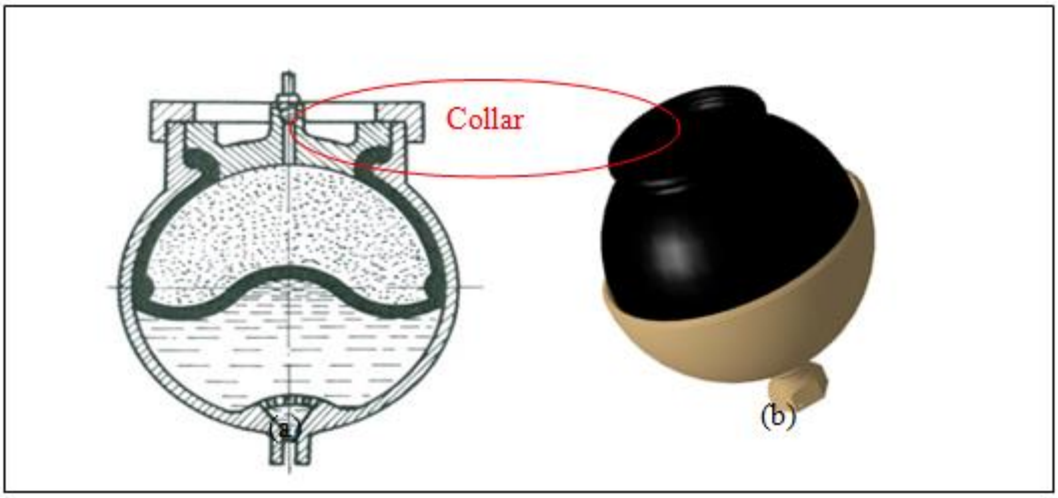

Figure 1: (a) & (b): Collar portion of Flexible Rubber of Diaphragm

Considering the various aspects of end use of flexible rubber diaphragm (FRD) and service failures due to nitrogen leakage through collar area, compression set requirement has been revised and reduced to 35% (max) in place of 60% (max) after taking inputs from ISO [9,10,12]as well as other national/international literature [14, page 7-14] without altering test conditions.

5.1.5 Other Physical-Mechanical Properties

Contents of existing technical specification [3] have been further studied and it is found that the other physico-mechanical properties specified such as swelling characteristics, brittleness & frost resistance temperature etc. are found appropriate and hence, there is no need for any changesin the qualification requirement. However, there is no end use specific properties have been specified. Considering the various aspects of end use of FRDs, requirement of such properties has

been discussed and deliberated at length and included in the newly evolved technical specification.

6. Technical Specification (requirement) Based on End Use Application

In the case of existing rubber compound, it appears that technical specification [3], of OEM14 is mainly for quality control checks and very much liberal. Such liberally specified requirements should not be mistaken to consider for development, evaluation and airworthiness certification of equivalent indigenous substitute rubber compound. It's not sufficient and needs to be tailored carefully to meet all end use specific technical requirements of FRD. There is a need to include other product oriented and end-use specific properties after considering various end use criticality of FRD as appended below:

6.1 Fatigue Properties:

The flexible rubber diaphragm fitted inside the spherical hydraulic accumulator of aircraft keeps on flexing continuously during the service exploitation. However, the extent of flexing and deformation depends on the fluctuation of pressure in the hydraulic system of aircraft. In view of ongoing and to establish the dynamic durability of flexible rubber diaphragm; it is pertinent to measure the fatigue properties by flexing technique, by measuring cut initiation & propagation resistance along with the heat built-up measurement etc.

The flexing properties of the rubbery materials are estimated by subjecting specified samples as per governing test specification and measure crack initiation and cut growth, after number of cyclic flexing. Heat built-up temperature on a specified specimen is also measured after number of cyclic and compressive loading, unloading at a regular interval of time.

6.2 Cut Initiation, Cut Growth and Heat Build-up:

On studying the requirement, specified in the technical specification of existing OEM14 material; it was observed that there is no such dynamic fatigue properties measurement stipulated.Whereas considering the severity of flexing of FRD undergoes during usage, it is imperative to specify dynamic fatigue properties to ascertain suitability. Considering the criticality of aircraft and non-specifying such important dynamic characteristics of OEM14; it was decided the estimate and include these properties in the new and evolved technical specification. In this regard, necessary inputs from various international/national specification and literature [12, 13, 15, 16, 17] for grading the flexible rubber diaphragm material and number of fatigue cycles for cut initiation, cut growth and heat buildup in comparison with OEM14 rubber compound have been decided to include for evaluation purpose.Accordingly, requirements have been captured and included in the newly evolved technical specification.

6.3 Air Permeability

The wall of flexible rubber diaphragm acts as a barrier between Nitrogen (N_2) at 5MPa and Hydraulic oil FH-51 at 21MPa (max.).Therefore, percolation of hydraulic oil into nitrogen cavity or vice versa is a cause of concerned. It will not only lead to mixing of two fluids but also loss of pressure and malfunction of entire hydraulic system of the aircraft. As RFD is spherical in shape, it acts as a pressurised device and absorbs the pressure impulse generated during hydraulic system actuation. It is pertinent to measure the gas permeability since the rubber diaphragm is always being subjected to pressure from both sides. There is no such air permeability properties measurement related information given in the existing specification. It was decided to estimate and include these properties in the new and evolved technical specification. In this regard, necessary inputs from ISO [12, page 111] for grading the flexible rubber diaphragm material have been decided.

Accordingly, requirements have been captured and included in the newly evolved technical specification.

6.4 Properties Retention in Fluid Medium

The degradation in mechanical properties after exposure in the working fluid needs to be ascertained to estimate the actual degradation taken place in the rubber material due to various reasons. The degradation is generally measured by measuring the loss in mechanical properties such as for change in tensile strength, elongation at break, and hardness. This change in properties, assist in tailoring the design of rubber compound and also helps in deciding the longevity of designed components made from particular grade of rubbery material. Therefore, the inclusion of measurement of loss in properties in the new technical specification is imperative.In this regard, necessary inputs from ISO [12, page 111] for grading the FRD rubber compound have been decided. Accordingly, requirements have been captured and included in the newly evolved technical specification.

6.5 Ozone Resistance

The existing rubber compound (OEM14)having nitrile rubber base polymer, which is un-saturate in nature and so the susceptible to degradation due to ozone.Moreover, it is imperative to impart moderate ozone resistance to the new design of rubber compound to overcome the degradation.There is no such requirement stipulated in existing specification (3). Therefore, inclusion of ozone exposure test have been stipulated and included in the technical requirements. In this regard, necessary inputs from ISO [12, page 111] for grading the material have been decided. Accordingly, requirements have been captured and included in the evolved technical specification.

6.6 Tear Strength

During service exploitation of aircraft, the FRDs undergoes sever deformation. The sever deformation is maximum experience at the outer surface of the central circumference portion of the diaphragm. Moreover, on studying the field failure data, it was observed that many flexible rubber diaphragms have also tear-off from the central portion as well as from other places. This property related requirement is not stipulated in existing specification. Therefore inclusion of measurement of tear strength test have been stipulated and included in comparison with existing OEM14 rubber compound. In this regard, necessary inputs from Atit Shah [9], Patent [10] and grading as per ISO [12, page 111] have been decided. Accordingly, requirements have been captured and included in the newly evolved technical specification.

7. EVOLVING OF NEW TECHNICAL SPECIFICATION AND GRAPHENE NANO PLATELETFILLED RUBBER-NANO-COMPOSITEDEVELOPMENT

Considering the various aspects of end use of flexible rubber diaphragm, stringent airworthiness certification requirements, analysis of service failure of RFD, new technical specification [18] evolved after incorporating the various properties. Accordingly, evaluation of indigenous development of graphene Nano platelet filled Rubber-Nano-Composite for aerospace application considered.

Accordingly, five formulations were designed and optimised to meet the entire qualification requirement. The designed formulations mainly consist of two grades of nitrile rubbers [containing 18 % acrylo-nitrile namely Perbunan 1846F with Mooney viscosity [ML (1+4) at 100 ^{0}C] 45and 28 % acrylo-nitrile namely Perbunan 2865F with Mooney viscosity [ML (1+4) at 100 ^{0}C] 70.Fast extrusion furnace (FEF) namely N550 and semi reinforcing furnace (SRF) namely N770 have been used for reinforcement of rubber matrix. Industrial quality graphene Nano-platelets (Grade GO4, thickness

11-15nm, surface area 60-80 m^2/g, diameter 5-15 microns, bulk density 0.03-0.10 g/cm^3). All the compounding ingredients were kept the same in all five designed formulation except varying graphene Nano platelet loading from 0 to 8 phr in the steps of 2 phr.The designed formulations identified as NPEG0, NPEG2, NPEG4, NPEG6 and NPEG8. The indigenous development was carried out in comparison with the existing OEM supplied rubber compound, OEM14. Graphene Nano-platelets varying in loading (i.e. from 0, 2, 4, 6 and 8 parts per hundred) have been included in the designed formulation to improve fatigue properties along with improvement in some end use performance of FRD. Other conventional compounding ingredients were used for protection from environmental conditions, ease and uniform dispersion of ingredients and appropriate curing system chosen for vulcanisation. Test results of newly designed rubber compounds were compared with the existing rubber compound OEM14. The compound codes and Graphene Nano platelets added in different compounds is given below: details of the formulation is given in table – 2

Table 2: phr loading of Graphene Nano platelets

Ingredient	NPEG0	NPEG2	NPEG4	NPEG6	NPEG8
Graphene Nano platelet	0.0	2.0	4.0	6.0	8.0

Other ingredients of the formulation are the same in phr for all formulations.

7.1Vulcanization:

The Rheometric properties of the compounds were tested at 145°C and tabulated in table 3A. Molding of different types of test samples related curing time is given in table 3A.

Table 3A: Curing characteristics obtained from Rheographsat 145^{0}C temperature

S No.	TestParameters	NPEG0	NPEG2	NPEG4	NPEG6	NPEG8	OEM14
1.	MH (lbs. inch)	58.84	57.22	55.13	54.72	55.80	68.83
2.	ML (lbs. inch)	5.34	5.86	6.36	5.81	5.92	15.11
3.	Tc90 (minutes)	8.40	10.33	11.38	8.22	9.14	9.92
4.	Ts_1 (minutes)	2.42	2.62	2.65	2.70	2.72	2.46
5.	Ts_2 (minutes)	2.59	2.79	2.81	2.87	2.83	2.83

The cure characteristics and ts1, ts2 of all the designed formulations are almost comparable with existing rubber compound OEM14. However; OEM14 has shown higher ML (initial torque) and MH (Maximum torque) in comparison with new designed formulations. It is mainly due to the fact that OEM14 is having hardness in the range of 76 to 79 Shore A (which is again due to comparatively high filler loading and little low plasticizer loading) whereas all new formulations designed with low hardness (i.e. 55±5 Shore A) requirement to enhance flexibility of FRD and meet end use service requirement.

Table 3B: Vulcanisation time for different types of Sample / Specimenat145^{0}C

S. No.	Particulars	NPEG0	NPEG2	NPEG4	NPEG6	NPEG8	OEM14
		Vulcanisation duration in minutes					
1.	Slab	11	13	14	11	12	12.5
2.	Flexing Strip	11	13	14	11	12	12.5
3.	Compression Set button	19	21	22	19	20	20.5
4.	Heat Build- up Button	19	21	22	19	20	20.5
Vulcanization duration for OEM-14 is as per existing manufacturing technology [19]: Temperature :143^{0}CDuration: 25 Minutes							

The vulcanised samples were free from visible defects.

7.2 Composition Analysis: Compositions of all the above mentioned rubber compound has been analysed using thermo-gravimetric analysis (TGA) and results reported in table 4.

Table 4: Composition Analysis by TGA (Test method: ASTM E-1131)

S No.	Particulars	NPEG0	NPEG2	NPEG4	NPEG6	NPEG8	OEM14
1.	Low boiling Material (%)	16.9	16.8	17.5	17.2	17.4	15.2
2.	Polymer content (%)	51.4	51.5	50.3	49.5	49.4	42.9
3.	Carbon black content (%)	28.3	28.3	28.2	28.4	28.5	37.9
4.	Ash Content (%)	03.4	03.2	03.8	04.9	04.7	03.9

It is observed from the contents of table 4 that; OEM14 rubber compound is higher carbon black loading and less polymer contents as compared to newly designed rubber compounds (.i.e. NPEG0, NPEG2, NPEG4, NPEG6, NPEG8). Higher filler loading with lower polymer content leads to higher hardness of OEM14. Higher hardness may be suitable for products like seals, rubber bonded valves etc. but definitely not suitable for products like FRD which undergoes sever flexing during service usage.

7.3 Graphene Nano platelet Suitability: Various properties specified in the new technical specification [18] were also measured in accordance with the test specifications. Measured results are reported in table 5.

Table 5: Measured Physical-Mechanical properties as per new evolved Technical Specification [18]

S No.	Particulars	Requirement	NPEG0	NPEG2	NPEG4	NPEG6	NPEG8	OEM14
1.	Tensile Strength (MPa) (*Gost 270-64)	11.0	13.7	14.9	13.9	14.7	14.5	13.4
2.	Elongation at break %	160	540	577	525	546	504	232
3a.	Modulus @100 % (MPa)	-	2.0	1.9	2.2	2.2	2.2	7.0
b	Modulus @200 % (MPa)	-	5.1	5.1	5.0	5.0	5.2	13.3
c.	Modulus @300 % (MPa)	-	8.1	8.3	8.0	8.1	8.3	-
4.	Residual Elongation at break (Max.)% (Gost 268-53)	8	8	10	6	12	11	4
5.	Hardness (Shore-A)	55±5	58	59	60	61	64	73
6.	Density, gm/m3 (Gost 267-60)	1.20 ± 0.05	1.18	1.18	1.18	1.19	1.19	1.28
7.	Volumetric Swelling in Oil FH-51 at 70^{0}C for 24 Hrs% (Ost 421-49)	4 to 10	3.46	2.89	5.56	4.96	6.95	10.7
8.	Relative Residual Deformation @30% compression/ 100°C / 72 Hrs, in Oil FH-51 (Max) % (TU-38-005-1166-73)	35	24.8	29.3	26.2	26.3	30.3	50.8
9.	Gravimetric Swelling in medium, for 24 Hrs (%)(Ost 421-49)							
a.	Mixtureof Iso-Octane + Toluene(70:30)at RT (Max)	25	17.0	15.8	16.3	16.1	16.4	
b.	In fuel T1 (ATFK-50) at RT	0 to 12	5.0	6.3	7.41	7.3	7.4	5.85
c.	In Oil FH-51 at 70^{0}C	-1 to 5	1.59	1.39	3.26	2.8	4.1	6.28

d.	In oil MS-20 at 130°C	-12	-11.6	-11.0	-9.8	-10	-11	-6.28

S No.	Particulars	Requirement	NPEG0	NPEG2	NPEG4	NPEG6	NPEG8	OEM14
10.	Change in % Elongation after ageing at 100^0C in air for 72 Hrs.							
a.	Change in(%)Elongation	-55 to -5	-26	-32	-22	-27	-28	-45
b.	Ageing Co-efficient	0.45 to 0.95	0.74	0.68	0.78	0.73	0.71	0.45
11.	Brittleness Temperature, °C (Max) (Gost 7912-56)	-38	Passes the test					
12.	Co-efficient of Frost resistance at -30°C, (Min) (Gost 13808-79)	0.15	0.4	0.43	0.45	0.45	0.43	0.4
13.	Ageing Co-efficient at 90°C in air for 21 days (Min)(Gost 271-67)	0.5	0.61	0.5	0.52	0.50	0.5	0.43
14.	After ageing in oil FH-15 at100^0C for 24							
a.	Change in Hardness(Shore A)(Max)	±10	-2	-3	-5	-4	-5.4	-6.6
b.	% Change in Tensile Strength Max	±15	-5.84	-11.4	-7.9	-13.6	-13.1	-10.7
c.	% Change in Elongation at break (Max)	±25	-16.5	-23.4	-16.6	-24.5	-20.6	-21.1
15.	Heat build-up (°C)	-	38	41	40	40	42	55
16.	De-Mattia Flexing-Cut Initiation (KCs) IS-3400(Pt.7)	-	32	43	82	50	46	14
17.	De-Mattia Flexing-Cut growth up to 12 mm (KCs) IS-3400(Pt.8)	-	3.5	6.9	7.3	6.8	5.1	180 cycles
18.	Air Permeability (m^2/Pa s)IS-3400(Pt.21)	-	6.6×10^{-17}	6.8×10^{-17}	6.0×10^{-17}	6.4×10^{-17}	6.4×10^{-17}	8.1×10^{-17}
19.	Tg by DSC (^{0}C) ASTMD-3418	-	-54.16	-52.60	-51.76	-55.27	-	-43.28& -37.96
20.	Ozone Resistance (25 pphm / RT / 20%Strain / 24 Hrs) (IS-3400 Pt.20)	No Cracks	Cracks observed after 24hrs.					Cracks observed within 24hrs.

Table -5 data shows that, indigenous rubber-nano-composite meeting almost entire new technical specification [18] requirements and having superior fatigue properties compared to OEM14.

8.5 Dynamic Mechanical Analysis

Dynamic properties of all the newly designed formulations of rubber Nano-composites were measured by using Mechanical Analysis (DMA) at room temperature, 0.5% static, 0.1% dynamic with 5Hz frequency and results are tabulated in Table 6.

Table 6: Dynamic Mechanical Analysis (DMA) properties

S No.	Particulars	NPEG0	NPEG2	NPEG4	NPEG6	NPEG8	OEM14
1.	Storage Modulus E' (MPa)	6.25	7.47	6.85	8.3		14.3
2.	Loss Modulus E"(MPa)	1.02	1.19	1.12	1.33		1.69
3.	Tan-delta	0.163	0.160	0.164	0.161		0.118
Test Method:ASTM D-5992							

Data shows that tan delta of new compounds is at slightly higher side than OEM14 compound, indicates more damping, dissipation of energy and reflecting in more cycles required for cut initiation and growth. Storage modulus (E') of OEM14 is quite high as compared to new designed rubber nano-composites. It may be due to higher cross linking of polymeric chains of OEM14, more filler content which increases more stiffness as well as higher hardness value.

9. CONCLUSIONS

A comprehensive study of the existing technical specification [3] of OEM14 revealed few shortfalls in the qualification requirement and appears to be for batch acceptance purpose only at manufacturer's site and should not be mistaken for development purpose. It is mainly since there is no specific mention and inclusions of end use and product oriented qualification test requirement, especially product like flexible rubber diaphragm (FRD). Moreover; some of the requirement specified is either higher side such as hardness, compression set and ageing coefficient etc. with wide acceptance limit or not at all included such as fatigue properties and tear strength etc.

Therefore, and in view of the above, it is imperative to evolve new technical specification in place of existing technical specification after considering the various aspect of Airworthiness Certification, service failure of flexible rubber diaphragm (FRD) and end use criticality of aircraft. Considering all aspects as discussed here, it was possible to evolve new technical specification [18] successfully. New technical specification is comprehensive and includes entire qualification requirement for Airworthiness Certification of rubber-nano-composite for Aerospace application.

REFERENCES

1. *hppts://en.wikipedia.org/List_of active_indian_military-aircraft. Retrieved on 11 Jan 2019*
2. *BS Mandloi, Regional Centre for Military Aircraft (Nasik), Defence Research and Development Organisation, Ministry of Defence C/o HAL (Aircraft Division) Ojhar Township: 422207. Compendium of indigenous substitutes for military aircraft. 2011*
3. *Technical Specification No. MPTU-38-00-1166: 1973,1987*
4. *RPKhapli et al. Study report on investigation of failure of diaphragm of spherical hydraulic accumulator of aircraft. Aircraft upgrade research and development centre, HAL Aircraft division, Nasik. TR/MiG-21/HYD/080. 21 Apr 2011*
5. *Development specification of indigenous rubber compound (used for manufacturing of seals, rubber bonded valves and flexible rubber diaphragm) No. SPEC/IND/NM/04: 5th Aug 2004*
6. *Service Bulletin No. 008109652-AB, 008-7-02254-4-AB; Major Items 66, 68, 69, 74, 75, 76, 77, 94, 94P, 96. Service life set up for major items, 21 of all modifications and their repair procedure: 21st Feb 1990*
7. *Minutes of 95thlifingcommittee meeting held in the office of Regional Director, RCMA (N) issued vide No. RCMA (N)/2000/F: 24th Jan 2001*

8. *Technical Specification No. TU No. 75-55-22: Rubber Diaphragm 76-5505-592*

9. *Atit Shah, Improving fatigue life for diaphragm: Neo-Tef diaphragm technology:Pump letter (www.neoflux.in): 19th August 2011.*

10. *Formula of rubber material of rubber diaphragm of high-temperature-resistant and low-temperature-resistant vacuum booster: Chinese patent CN102061037B: 13thMar 2013*

11. *RC Moakes, long term natural ageing report, Part 2-rubber properties (other than compression set) up to 15 Years. RAPARA Members Journal: July/Aug 1975*

12. *ISO 1433-1984 (E).ISO Standards handbook 22 (page 111). ISBN 92 67 10097 1(volume 2) (rubber mixes and vulcanised rubber):1984*

13. *ISO 11346-2014. Rubber, vulcanized or thermoplastic. Estimation of life time and maximum temperature of use: 2014*

14. *Roger P. Brown, Rubber Product Failure (page 7-14). ISBN 1-85957-330-4. Rapra technology limited, Shaw bury, Shrewsbury, Shropshire SY4 4NR, United Kingdom: 2001*

15. *WV Mars et al. A literature survey on fatigue analysis approaches for rubber. International journal of fatigue 24 (2002) 949–961. Elsevier Ltd.: 17 Dec 2001*

16. *C. Cruanes et al. Study of the fatigue behaviour of a synthetic rubber undergoing cumulative damage tests. International journal of fatigue 91 (2016) 322–327.Elsevier Ltd.: 5 Dec 2015*

17. *WD Kim et al. Fatigue life estimation of an engine rubber mount. International journal of fatigue 26 (2004) 553–560.Elsevier Ltd.: 14 Aug 2003*

18. *Development specification of indigenous rubber compound (used for manufacturing of rubber diaphragm use in hydraulic accumulator) No. SPEC/IND/NM/16: 16Aug 2008*

19. *Flexible rubber diaphragm drawing No. 76-5505-592: 21stApl 1994*

20. *Ramaswamy, R., et al. "Investigation of densification behaviour on yttrium oxide reinforced Ti-6Al-4V nano-composite through powder metallurgy." Int J Mech Prod Eng Res Dev 8.2: 433-442.*

21. *Kook Chan Ahn& Ho Duck Kang, "Stress Characteristics of Nanocomposite Laminates with Effect of Nanoclay Content", International Journal of Mechanical and Production Engineering Research and Development (IJMPERD), Vol. 9, Issue 6, pp, 653–660.*

22. *M. El-Shennawy& Adel A. Omar, "Manufacturing of Nano/Micro Composites using Friction Stir Processing", International Journal of Mechanical Engineering (IJME), Vol. 4, Issue 3, pp, 29-48.*

23. *AzeemPasha& B.M. Rajaprakash, "Hardness And Microstructure Of Functionally Graded Aluminium Silicon Alloy With Multiwall Carbon Nanotube Composite By Powder Metallurgy With Hot Extrusion Technique", International Journal of Mechanical and Production Engineering Research and Development (IJMPERD), Vol. 10, Issue 3, pp, 12279–12288*